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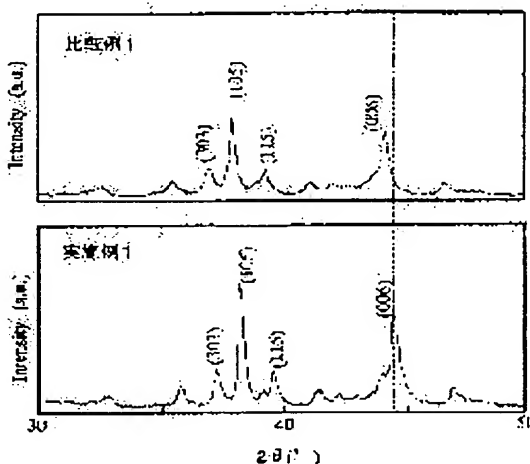
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(54) R-T-B-C BASED SINTERED MAGNET AND PRODUCTION METHOD THEREFOR

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a high performance R-T-B-C based sintered magnet (wherein R is at least one rare earth element; and T is Fe, or Fe and Co), and a production method therefor.

SOLUTION: An R-T-B-C based alloy having the main componential composition of, by weight, 28 to 33% R (wherein R is at least one rare earth element) and 0.9 to 1.1% B+C (wherein the content of B is 0.6 to 0.9%, and the content of C is 0.15 to 0.3) and the balance T (wherein T is Fe, or Fe and Co) and having a main phase consisting of an R₂T₁₄ (B, C) phase is pulverized. The obtained fine powder is recovered into a nonoxidizing solution consisting of oil such as mineral oil and at least one kind of lubricant selected from the monovalent alcohol ester of a polybasic acid, the fatty acid ester of polyhydric alcohol, and their derivatives, and is then subjected to forming, degreasing, sintering, and heat treatment.



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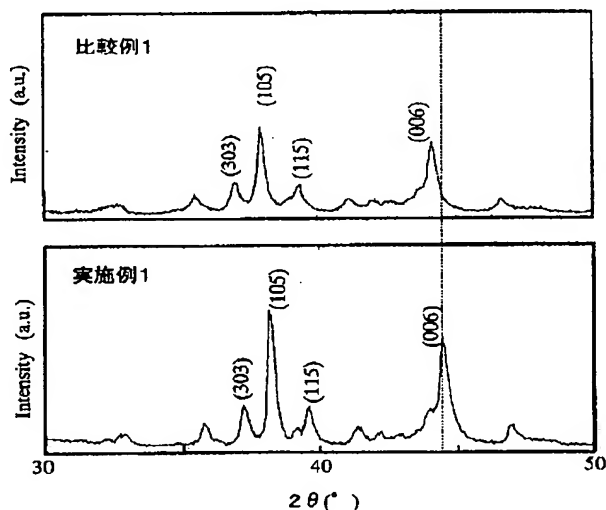
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(54) 【発明の名称】 R-T-B-C系焼結磁石及びその製造方法

(57) 【要約】

【課題】 高性能R-T-B-C系焼結磁石（但しRは希土類元素の少なくとも1種であり、TはFe、又はFe及びCoである）及びその製造方法を提供する。

【解決手段】 重量%でR（但しRは希土類元素の少なくとも1種である）：28～33%、B+C：0.9～1.1%（但しB：0.6～0.9%であり、C：0.15～0.3%である）、及び残部：T（但しTはFe、又はFe及びCoである）の主要成分組成を有し、R₂T₁₄（B、C）相を主相とするR-T-B-C系合金を微粉砕し、得られた微粉を鉱油等の油と、多塩基酸の1価アルコールエステル、多価アルコールの脂肪酸エステル及びそれらの誘導体から選択される少なくとも1種の潤滑剤とからなる非酸化性液中に回収し、次いで成形、脱油、焼結、熱処理することを特徴とするR-T-B-C系焼結磁石の製造方法。



【特許請求の範囲】

【請求項 1】 重量%で R（但し R は希土類元素の少なくとも 1 種である）：28～33%、B + C：0.9～1.1%

（但し B：0.6～0.9%であり、C：0.15～0.3%である）、及び残部：T（但し T は Fe、又は Fe 及び Co である）の主要成分組成を有し、 R_2T_{14} （B、C）相を主相とする R-T-B-C 系合金を非酸化性雰囲気中で平均粒径 1～10 μ m に微粉砕し、得られた微粉を鉱油、合成油及び植物油から選択される少なくとも 1 種の油と、多塩基酸の 1 価アルコールエステル、多価アルコールの脂肪酸エステル及びそれらの誘導体から選択される少なくとも 1 種の潤滑剤とからなる非酸化性液中に回収してスラリー化し、次いで前記スラリーを成形し、得られた成形体を脱油し、焼結し、熱処理することを特徴とする R-T-B-C 系統結磁石の製造方法。

【請求項 2】 鉱油、合成油及び植物油から選択される少なくとも 1 種の油と、多塩基酸の 1 価アルコールエステル、多価アルコールの脂肪酸エステル及びそれらの誘導体から選択される少なくとも 1 種の潤滑剤との混合比率が 99.7～99.99 重量部：0.3～0.01 重量部である請求項 1 に記載の R-T-B-C 系統結磁石の製造方法。

【請求項 3】 重量%で R（但し R は希土類元素の少なくとも 1 種である）：28～33%、B + C：0.9～1.1%（但し B：0.6～0.9%であり、C：0.15～0.3%である）、及び残部：T（但し T は Fe、又は Fe 及び Co である）の主要成分組成を有する合金溶湯をストリップキャスト法により凝固し、 R_2T_{14} （B、C）主相及び R リッチ相から実質的になるとともに前記主相の短軸方向の平均結晶粒径が 3～20 μ m である板厚 0.05～3 mm の R-T-B-C 系合金を得、次にこの合金を粗粉化し、次いで非酸化性雰囲気中で平均粒径 1～10 μ m に微粉砕し、得られた微粉を鉱油、合成油及び植物油から選択される少なくとも 1 種の油と、多塩基酸の 1 価アルコールエステル、多価アルコールの脂肪酸エステル及びそれらの誘導体から選択される少なくとも 1 種の潤滑剤とからなる非酸化性液中に回収してスラリー化し、次いで前記スラリーを成形し、得られた成形体を脱油し、焼結し、熱処理することを特徴とする R-T-B-C 系統結磁石の製造方法。

【請求項 4】 鉱油、合成油及び植物油から選択される少なくとも 1 種の油と、多塩基酸の 1 価アルコールエステル、多価アルコールの脂肪酸エステル及びそれらの誘導体から選択される少なくとも 1 種の潤滑剤との混合比率が 99.7～99.99 重量部：0.3～0.01 重量部である請求項 3 に記載の R-T-B-C 系統結磁石の製造方法。

【請求項 5】 ストリップキャスト法により凝固して得られた R-T-B-C 系合金を 800～1100℃で熱処理し、次いで粗粉化する請求項 3 又は 4 に記載の R-T-B-C 系統結磁石の製造方法。

【請求項 6】 重量%で R（但し R は希土類元素の少な

くとも 1 種である）：28～33%、B + C：0.9～1.1%（但し B：0.6～0.9%であり、C：0.15～0.3%である）、及び残部：T（但し T は Fe、又は Fe 及び Co である）の主要成分組成を有し、 R_2T_{14} （B、C）相を主相とすることを特徴とする R-T-B-C 系統結磁石。

【請求項 7】 R_2T_{14} （B、C）主相の格子定数比：c/a（ただし、c は正方晶の一軸異方性方向の格子定数であり、a は残りの 2 辺の格子定数である）が 1.375～1.385 である請求項 6 に記載の R-T-B-C 系統結磁石。

【請求項 8】 重量%で R：28～33%、B + C：0.9～1.1%（但し B：0.6～0.9%であり、C：0.15～0.3%である）、M：0.01～0.3%（但し M は Cu、Al、Ga、Nb 及び Mn からなる群から選択される少なくとも 1 種である）及び残部：T（但し T は Fe 及び Co であり、Co：0.5～5%である）の主要成分組成を有する請求項 6 又は 7 に記載の R-T-B-C 系統結磁石。

【請求項 9】 重量%で R（但し R は希土類元素の少なくとも 1 種である）：28～32%、B + C：0.9～1.1%（但し B：0.6～0.9%であり、C：0.15～0.3%である）、及び残部：T（但し T は Fe、又は Fe 及び Co である）の主要成分組成を有し、 R_2T_{14} （B、C）主相の格子定数比：c/a（ただし、c は正方晶の一軸異方性方向の格子定数であり、a は残りの 2 辺の格子定数である）が 1.375～1.385 である R-T-B-C 系統結磁石であって、前記 R-T-B-C 系統結磁石の単位重量あたりの含有酸素量が 0.3 重量%以下であり、かつ焼結体密度が 7.56 Mg/m³ 以上であることを特徴とする R-T-B-C 系統結磁石。

【請求項 10】 重量%で R（但し R は希土類元素の少なくとも 2 種であって Nd 及び Dy を必須に含み、Dy 含有量が 0.3～15%である）：28～32%、B + C：0.9～1.1%（但し B：0.6～0.9%であり、C：0.15～0.3%である）、及び残部：T（但し T は Fe、又は Fe 及び Co である）の主要成分組成を有し、 R_2T_{14} （B、C）主相の格子定数比：c/a（但し c は正方晶の一軸異方性方向の格子定数であり、a は残りの 2 辺の格子定数である）が 1.375～1.385 である R-T-B-C 系統結磁石であって、前記希土類焼結磁石の単位重量あたりの含有酸素量が 0.3 重量%以下であり、かつ焼結体密度が 7.60 Mg/m³ 以上であることを特徴とする R-T-B-C 系統結磁石。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、R-T-B-C 系合金（但し R は希土類元素の少なくとも 1 種であり、T は Fe、又は Fe 及び Co である）からなる焼結磁石及びその製造方法に関する。

【0002】

【従来の技術】Nd-Fe-B系焼結磁石は、所定組成のNd-Fe-B系合金を粗粉碎し、次いでN₂等の不活性ガス中で微粉碎し、得られた平均粒径1~10 μ mの微粉末を磁場中成形し、焼結し、熱処理することにより製造され、各種磁石応用製品分野で多用されている。

【0003】Nd-Fe-B系焼結磁石の磁気特性を高めるために、一般的に不可避免の不純物元素であるCの含有量は極低いことがよいとされている。特許第2731337号公報には、Nd-Fe-B系焼結磁石用原料微粉末と鉱油又は合成油とを混合してスラリー化し、次いで湿式成形し、得られた成形体を脱油し、焼結し、熱処理する高性能Nd-Fe-B系焼結磁石の製造方法が開示されている。この方法によると従来に比べて酸素含有量が顕著に低減されるとともに焼結性が向上し、高い残留磁束密度Br、高い最大エネルギー積(BH)_{max}、及び高い固有保磁力iHcを有するNd-Fe-B系焼結磁石を製造することができる。これらの高い磁気特性は、成形体中に上記油分が極力残留しないように焼結の前に脱油(脱C)処理を行い、最終的に得られるNd-Fe-B系焼結磁石のC含有量を0.1重量%以下に低減することによって達成されている。

【0004】特開平9-17677号公報は、R(但しRはYを含む希土類元素の少なくとも1種である)12~18原子%、B+C=6~10原子%(但しB:2~6原子%、C:4~8原子%である)、残部Fe(但しFeの1部をCo、Niの1種又は2種にて置換できる)及び不可避免の不純物からなる合金溶湯を、ストリップキャスティング法にて板厚0.03~10mmの薄板で、Rリッチ相が10 μ m以下に微細に分離した組織を有する鋳片に铸造後、該鋳片を粗粉碎して得た平均粒度10~500 μ mの粗粉碎粉に液状潤滑剤又は固状潤滑剤を0.02~5.0重量%添加混合して微粉碎し、得られた平均粒径1~10 μ mの微粉末をモールド内に充填密度1.4~3.5Mg/m³に充填し、瞬間的に795.8kA/m(10kOe)以上のパルス磁場をかけて配向後、成形、焼結、時効処理することにより耐食性のすぐれたR-Fe-B-C系永久磁石材料を製造する方法を開示している。しかし、この永久磁石材料はC:4~8原子%という高C含有量の点で本発明の焼結磁石とは組成が異なる。具体例を挙げれば、特開平9-17677号公報の表1の組成1の永久磁石はNd:12.8原子%、Dy:1.5原子%、Co:10原子%、B:3.2原子%、C:4.4原子%、及びFe:68.1原子%(Nd:28.2重量%、Dy:3.7重量%、Co:9.0重量%、B:0.5重量%、C:0.8重量%、及びFe:57.9重量%)の組成を有している。又本発明者らの検討から、R₂T₁₄(B、C)主相は酸化されやすく、もって酸化によりR₂T₁₄(B、C)主相比率が低下し、室温において安定して350.1kJ/m³(44MG0e)以上の(BH)_{max}でかつ1.1MA/m(14kOe)以上のiHcを得られないことがわかった。

【0005】特許第2739502号公報は、R-Fe-B-C系合金磁石(但しRはYを含む希土類元素の少なくとも1種である)において、該合金の磁性結晶粒の各々が耐酸化性保護膜で覆われており、この耐酸化性保護膜は該磁性結晶粒を構成している合金元素の実質上全てを含みかつその0.1~16原子%がCである耐酸化性の優れたものを開示している。しかし、本発明の焼結磁石と比較し、この公報の各実施例のR-Fe-B-C系合金磁石は高R-低B-高C組成であつたり、又は低R-低B-高C組成のものである。具体例を挙げれば、特許第2739502号公報の実施例1の磁石組成はNd:18原子%、Fe:71原子%、B:1原子%、及びC:10原子%(Nd:38.8重量%、Fe:59.3重量%、B:0.1重量%、及びC:1.8重量%)であり、実施例11の磁石組成はNd:10原子%、Fe:79原子%、B:1原子%、及びC:10原子%(Nd:24.1重量%、Fe:73.7重量%、B:0.2重量%、及びC:2.0重量%)である。さらに得られている磁気特性は非常に低く、又本発明者らの検討から、これらの組成を選択した場合、室温において350.1kJ/m³(44MG0e)以上の(BH)_{max}で、かつ1.1MA/m(14kOe)以上のiHcを得られないことがわかった。

【0006】

【発明が解決しようとする課題】したがって本発明が解決しようとする課題は、主要成分として所定量のCを含有した高C含有型であっても高い磁気特性を有するR-T-B-C系焼結磁石(但しRは希土類元素の少なくとも1種であり、TはFe、又はFe及びCoである)及びその製造方法を提供することである。

【0007】

【課題を解決するための手段】上記課題を解決した本発明のR-T-B-C系焼結磁石の製造方法は、重量%でR(但しRは希土類元素の少なくとも1種である):28~33%、B+C:0.9~1.1%(但しB:0.6~0.9%であり、C:0.15~0.3%である)、及び残部:T(但しTはFe、又はFe及びCoである)の主要成分組成を有し、R₂T₁₄(B、C)相を主相とするR-T-B-C系合金を非酸化性雰囲気中で平均粒径1~10 μ mに微粉碎し、得られた微粉を鉱油、合成油及び植物油から選択される少なくとも1種の油と、多塩基酸の1価アルコールエステル、多価アルコールの脂肪酸エステル及びそれらの誘導体から選択される少なくとも1種の潤滑剤とからなる非酸化性液中に回収してスラリー化し、次いで前記スラリーを成形し、得られた成形体を脱油し、焼結し、熱処理することを中心とする。本発明のR-T-B-C系焼結磁石の製造に際し、Cの添加時期は合金の溶解段階か、あるいは溶解以前の段階(溶解用原料中にあらかじめ所定量のCを含有させておく)とする必要がある。この理由は不可避免の不純物レベルのCを含有するR₂T₁₄B相を主相とするR-T-B系合金を溶製後、例えばカーボンブラック等の形で粗粉碎時又は微粉碎時

に所定量のCを添加し、次いで磁場中成形し、焼結し、熱処理してなる焼結磁石では、粗粉碎時又は微粉碎時に添加したCが $R_2T_{14}B$ 主相中へほとんどとり込まれず、Rリッチ相中にCが濃縮される傾向が顕著であり、 $R_2T_{14}(B, C)$ 相による磁気特性の向上効果を期待できないからである。

【0008】又本発明のR-T-B-C系統結磁石の製造方法は、重量%でR（但しRは希土類元素の少なくとも1種である）：28~33%、B+C：0.9~1.1%（但しB：0.6~0.9%であり、C：0.15~0.3%である）、及び残部：T（但しTはFe、又はFe及びCoである）の主要成分組成を有する合金溶湯をストリップキャスト法により凝固し、 $R_2T_{14}(B, C)$ 主相及びRリッチ相から実質的になるとともに前記主相の短軸方向の平均結晶粒径が3~20 μm である板厚0.05~3mmのR-T-B-C系合金を得、次にこの合金を粗粉化し、次いで非酸化性雰囲気中で平均粒径1~10 μm に微粉碎し、得られた微粉を鉱油、合成油及び植物油から選択される少なくとも1種の油と、多塩基酸の1価アルコールエステル、多価アルコールの脂肪酸エステル及びそれらの誘導体から選択される少なくとも1種の潤滑剤とからなる非酸化性液中に回収してスラリー化し、次いで前記スラリーを成形し、得られた成形体を脱油し、焼結し、熱処理することを特徴とする。

【0009】本発明のR-T-B-C系統結磁石の製造方法において、鉱油、合成油及び植物油から選択される少なくとも1種の油と、多塩基酸の1価アルコールエステル、多価アルコールの脂肪酸エステル及びそれらの誘導体から選択される少なくとも1種の潤滑剤との混合比率を99.7~99.99重量部：0.3~0.01重量部とすると成形体の配向度が向上するので好ましく、もってBr及び(BH)maxが向上する。混合比率が前記特定範囲を外れると成形体の配向度向上効果を得られなかったり、あるいは粒界相に納入されるC量が増大してRが希土類炭化物を形成し、焼結不良を招来する。

【0010】本発明のR-T-B-C系統結磁石の製造方法において、ストリップキャストして得られたR-T-B-C系合金を実質的な真空中あるいは不活性ガス雰囲気中において800~1100℃で熱処理し、次いで粗粉化し、微粉碎すると粒径分布のシャープな微粉になり、もって最終的に得られるR-T-B-C系統結磁石のBr、(BH)max及び減磁曲線の角形性を向上することができ

る。

【0011】本発明のR-T-B-C系統結磁石は、重量%でR（但しRは希土類元素の少なくとも1種である）：28~33%、B+C：0.9~1.1%（但しB：0.6~0.9%であり、C：0.15~0.3%である）、及び残部：T（但しTはFe、又はFe及びCoである）の主要成分組成を有し、 $R_2T_{14}(B, C)$ 相を主相とすることを特徴とする。本発明のR-T-B-C系統結磁石にお

いて、 $R_2T_{14}(B, C)$ 主相の格子定数比： $c/a = 1.375 \sim 1.385$ （ただし、 c は正方晶の一軸異方性方向の格子定数であり、 a は残りの2辺の格子定数である）のときに高いBr及び(BH)maxを得られる。又本発明のR-T-B-C系統結磁石において、重量%でR：28~33%、B+C：0.9~1.1%（但しB：0.6~0.9%であり、C：0.15~0.3%である）、M：0.01~0.3%（但しMはCu、Al、Ga、Nb及びMnからなる群から選択される少なくとも1種である）及び残部：T（但しTはFe及びCoであり、Co：0.5~5%である）の主要成分組成を選択すると磁気特性や、耐食性を向上できるので好ましい。

【0012】又本発明のR-T-B-C系統結磁石は、重量%でR（但しRは希土類元素の少なくとも1種である）：28~32%、B+C：0.9~1.1%（但しB：0.6~0.9%であり、C：0.15~0.3%である）、及び残部：T（但しTはFe、又はFe及びCoである）の主要成分組成を有し、 $R_2T_{14}(B, C)$ 主相の格子定数比： c/a （ただし、 c は正方晶の一軸異方性方向の格子定数であり、 a は残りの2辺の格子定数である）が1.375~1.385であるR-T-B-C系統結磁石であって、前記R-T-B-C系統結磁石の単位重量あたりの含有酸素量が0.3重量%以下であり、かつ焼結体密度が7.56Mg/m³以上であることを特徴とする。

【0013】又本発明のR-T-B-C系統結磁石は、重量%でR（但しRは希土類元素の少なくとも2種であってNd及びDyを必須に含み、Dy含有量が0.3~15%である）：28~32%、B+C：0.9~1.1%（但しB：0.6~0.9%であり、C：0.15~0.3%である）、及び残部：T（但しTはFe、又はFe及びCoである）の主要成分組成を有し、 $R_2T_{14}(B, C)$ 主相の格子定数比： c/a （但し c は正方晶の一軸異方性方向の格子定数であり、 a は残りの2辺の格子定数である）が1.375~1.385であるR-T-B-C系統結磁石であって、前記希土類焼結磁石の単位重量あたりに含有される酸素量が0.3重量%以下であり、かつ焼結体密度が7.60Mg/m³以上であることを特徴とする。

【0014】本発明のR-T-B-C系統結磁石において、R成分の酸化物化及び炭化物化を抑制してR成分に富んだ粒界相を確保し、 $R_2T_{14}(B, C)$ 主相比率を極力高め、もってBr、(BH)max及びiHcを高めるために、含有酸素量を0.2重量%以下にするのがより好ましく、0.18重量%以下にするのがさらに好ましい。

【0015】

【発明の実施の形態】本発明のR-T-B-C系統結磁石の組成限定理由を以下に説明する。以下単に%と記すのは重量%を意味するものとする。

【0016】R量は28~33%が好ましい。良好な耐食性を具備し、(BH)maxを高めるために、R量は28~32%がより好ましく、28~31%が特に好ましい。R量が28%未

満では実用に耐える*iHc*を得られず、33%超ではBr、(BH)_{max}の低下が顕著になる。不可避的R成分を除いて、 $R = Nd + Dy$ 、 $Nd + Dy + Pr$ 、 $Nd + Pr$ 、又は $Pr + Dy$ の場合の実用性が高い。実用に耐える保磁力(耐熱性)を付与するために、Dy含有量は0.3~10%が好ましく、0.5~8%にするのがより好ましい。Dy含有量が0.3%未満では保磁力の向上効果を得られず、10%超ではBr、(BH)_{max}が大きく低下する。

【0017】B及びCは必須元素であり、(B+C)量は0.9~1.1%が好ましく、0.95~1.1%がより好ましい。B量は0.6~0.9%が好ましく、0.65~0.85%がより好ましい。C量は0.15~0.3%が好ましく、0.18~0.28%がより好ましい。B量が0.6%未満ではたとえC量が上記特定含有量範囲内でもR₂Fe₁₇金属間化合物の生成を伴い、Br及び*iHc*が大きく低下する。又B量が0.9%超ではCが主相中にほとんど入る余地がなくなり、粒界相に不純物として濃縮されるのでBr及び*iHc*が大きく低下する。C量が0.15%未満ではR₂Ti₄(B、C)相の生成量が過少なためR₂Ti₄(B、C)相による磁気特性向上効果を事実上得られず、0.3%超では粒界相にCが濃縮され、焼結不良を招来するとともに磁気特性が急激に低下する。即ち、上記特定(B+C)量範囲のときにR₂Ti₄(B、C)相が主相として生成し、もって従来の高C含有型Nd-Fe-B焼結磁石では得られなかった高い磁気特性を得ることができる。

【0018】Cu、Al、Ga、Nb及びMnからなる群から選択される少なくとも1種の元素Mを0.01~0.3%含有することにより磁気特性や、耐食性を高めることができる。Alを0.01~0.3%含有することにより*iHc*が向上し、耐食性が改善される。しかし、Al含有量が0.3%超ではBrの低下が顕著になり、0.01%未満では*iHc*及び耐食性を高める効果を得られない。より好ましいAl含有量は0.05~0.3%である。Gaを0.01~0.3%含有することにより*iHc*が顕著に向上するが、Ga含有量が0.3%超ではBrの低下が大きくなり、0.01%未満では*iHc*を高める効果を得られない。より好ましいGa含有量は0.05~0.3%である。Cuを0.01~0.3%含有することにより耐食性及び*iHc*が向上するが、Cu含有量が0.3%超ではBrの低下が顕著になり、0.01%未満では耐食性及び*iHc*を高める効果を得られない。より好ましいCu含有量は0.05~0.3%である。

【0019】Coを0.5~5%含有することにより耐食性が向上し、キュリー点が上昇し、もって耐熱性が向上する。しかしCo含有量が5%超では磁気特性に有害なFe-Co相が生成し、Br及び*iHc*が大きく低下する。又Co含有量が0.5%未満では耐食性及び耐熱性を向上する効果を得られない。又Coを0.5~5%及びCuを0.01~0.3%含有するときに1.1MA/m(14kOe)以上の室温の*iHc*を得られる第2次熱処理温度の許容範囲が広がる効果を得られ、特に好ましい。

【0020】不可避に含まれる酸素量は0.3%以下が好ましく、0.2%以下がより好ましく、0.18%以下が特に好ましい。酸素含有量を0.3%以下に低減することによりR₂Ti₄(B、C)主相比率が高まり、焼結体密度を略理論密度まで高めることができる結果、室温において350.1kJ/m³(44MGOe)以上の(BH)_{max}で、かつ1.1MA/m(14kOe)以上の*iHc*を安定して得ることができる。焼結体密度は例えばNd-Pr-T-B-C系焼結磁石で7.56Mg/m³以上になり、Nd-Dy-T-B-C系焼結磁石で7.60Mg/m³以上になる。又良好な耐食性及び高い磁気特性を具備するために、不可避に含まれる窒素量を0.15%以下にするのが好ましく、0.002~0.15%にするのがより好ましい。窒素量が0.15%を超えるとBrの低下が顕著になる。

【0021】本発明のR-T-B-C系焼結磁石は例えば以下のようにして製造できる。まず、所定組成に調整したR-T-B-C系合金溶湯を単ロール法又は双ロール法によりストリップキャストし、板厚:0.05~3mmの薄板でかつR₂Ti₄(B、C)主相と微細なRリッチ相とから実質的になるR-T-B-C系合金を得る。この合金中のR₂Ti₄(B、C)主相の短軸方向の平均結晶粒径は3~20μm程度であり、αFeのない組織であり、もって高い磁気特性のR-T-B-C系焼結磁石を得ることができる。必要に応じて前記薄板合金を不活性ガス雰囲気中で800~1100℃×0.5~10時間加熱する熱処理を行うことが好ましい。この熱処理により、微粉砕の粒径分布がシャープになり、Br及び(BH)_{max}を高めることができる。なお、熱処理条件が800℃×0.5時間未満では熱処理の効果が認められず、1100℃×10時間超では酸化による組成ずれ等の問題を生じる。前記薄板状R-T-B-C系合金中のCは主相中に取り込まれている。これはR-T-B-C系合金の溶解温度から室温に冷却されるまでの過程でR₂Ti₄Cが安定に形成する800℃~1100℃の温度領域を経て、CがR₂Ti₄B相中に取り込まれるからである。即ちR₂Ti₄Bの化学量論組成対比でBがやや不足している組成に調整したR-T-B系合金溶湯中に適量のCを存在させると、R₂Ti₄(B、C)相が生成され、高い磁気特性を発現すると考えられる。次に、前記薄板状R-T-B-C系合金に水素を吸蔵させて自然崩壊させ、次いで脱水素処理を行った後これを粗粉化する。次いで微粉砕する。微粉砕は不活性ガスを粉砕媒体とするジェットミルにより、例えば酸素濃度が0.1体積%未満、より好ましくは0.01体積%以下の不活性ガス雰囲気中で平均粒径1~10μmに微粉砕する。こうして得られた微粉を鉱油、合成油及び植物油から選択される少なくとも1種の油と、多塩基酸の1価アルコールエステル、多価アルコールの脂肪酸エステル及びそれらの誘導体から選択される少なくとも1種の潤滑剤とからなる非酸化性液中に回収してスラリー化する。次いで前記スラリーを磁場中成形し、得られ

た成形体を脱油し、焼結し、熱処理する。前記微粉の平均粒径は1~10 μ mが好ましく、3~6 μ mがより好ましい。平均粒径が1 μ m未満では粉碎効率が大きく低下し、10 μ m超ではiHc及びBrの低下が顕著になる。成形体の酸化による磁気特性の劣化を阻止するために、成形直後から脱油までの間前記油中で成形体を保存するのが望ましい。成形体を室温から焼結温度まで急激に昇温すると成形体の内部温度が急激に上昇し、成形体に残留する油と成形体を構成する希土類元素とが反応して希土類炭化物を生成し磁気特性が劣化する。この対策として、温度100~500℃、真空度13.3Pa (10⁻¹ Torr) 以下で30分間以上加熱する脱油処理を施すことが望ましい。脱油処理により成形体中の油が十分に除去される。なお、脱油処理の加熱温度は100~500℃であれば1点である必要はなく2点以上でもよい。また13.3Pa (10⁻¹ Torr) 以下で室温から500℃までの昇温速度を10℃/分以下、より好ましくは5℃/分以下とする脱油処理を施すことによっても脱油を効率よく行うことができる。鉱油、合成油又は植物油として、脱油及び成形性の点から、分留点が350℃以下のものがよい。又室温の動粘度が10cSt以下のものがよく、5cSt以下のものがさらに好ましい。

【0022】本発明のR-T-B-C系焼結磁石に対し、CuK α 線を線源として2 θ / θ 法により2 θ =30~50°の範囲でX線回折すると、(105)面と(006)面からの強いX線回折ピークを得られる。これら2本のX線回折ピーク位置(2 θ)から求めた面間隔d (d=X線波長/(2sine))と面指数とを式(1)に代入し、R₂T₁₄B相(正方晶)の格子定数aとcを求めることができる。

$$1/d^2 = (h^2 + k^2) / a^2 + l^2 / c^2 \quad (1)$$

【0023】

【実施例】以下、実施例により本発明を詳細に説明するが、それら実施例により本発明が限定されるものではない。

(実施例1) 重量%で、Nd:23.90%, Pr:6.60%, B:0.80%, C:0.18%, Co:2.00%, Ga:0.10%, Cu:0.10%及び残部:Feの主要成分組成を有し、板厚約0.3mm、短軸方向の平均結晶粒径が3 μ mのストリップキャスト合金を粗粉化し、次いで酸素濃度約1ppm(体積比)に調整した窒素ガス雰囲気中でジェットミル微粉碎した。得られた平均粒径4.0 μ mの微粉をこの窒素ガス雰囲気中で大気に触れることなく鉱油(出光興産(株)製、商品名:出光スーパーゾルPA-30)中に回収しスラリー化した。平均粒径はSympatec社製レーザー回折型粒径分布測定装置(商品名:ヘロス・ロードス)により測定した。次にスラリーに所定量のオレイン酸メチルを添加し、攪拌機により混合した。スラ

リーの配合内訳は、前記微粉:70重量部、鉱油:29.9重量部、オレイン酸メチル:0.10重量部とした。次にスラリーを圧縮成形用金型のキャビティに注入し、次いで配向磁場強度:1.0MA/m(13kOe)、及び成形圧力:98MPa(1.0ton/cm²)の条件で横磁場圧縮成形し、15mm×25mm×10mmの直方体状成形体を得た。次に、成形体を真空度約66.5Pa(5×10⁻¹ Torr)、200℃の条件で3時間加熱して脱油し、次いで同雰囲気中で1050℃まで昇温し、次いで1050℃で2時間焼結し、その後室温まで冷却した。次に焼結体をアルゴン雰囲気中で900℃で2時間加熱し、次いで室温まで急冷する第1次熱処理を行った。次にアルゴン雰囲気中で500℃で1時間加熱し、次いで室温まで冷却する第2次熱処理を行い本発明の焼結磁石を得た。得られた焼結磁石の組成分析結果を表1に示す。又得られた焼結磁石を10mm角に加工し、密度を測定した後、室温(20℃)の磁気特性を測定した結果を表2に示す。又得られた焼結磁石を7mm角に加工し、室温(20℃)において11.9MA/m(150kOe)のパルス磁場を印加し、磁化測定を行った。この測定によりBr及び磁化の最大値(4 π I)_{max}を求め、算出したB_r/(4 π I)_{max}=97.2%であった。又得られた焼結磁石に対し、以下の要領でX線回折(線源:CuK α 線)を行った。異方性付与方向に垂直な面が測定面となるように得られた焼結磁石をセットし、2 θ / θ 法により2 θ =30~50°を走査した。結果を図1に示す。いずれもNd₂Fe₁₄B相と同様のX線回折パターンのみが認められた。後述する比較例1のX線回折パターン(図1中)との対比から、実施例1の焼結磁石の(006)面のX線回折ピークが高角度側にシフトしていることがわかる。これは添加したCが主相中に存在していることを示す証拠であり、図1中の回折パターンは(Nd, Pr)₂(Fe, Co)₁₄(B, C)相を表している。(006)面と(105)面の回折ピーク位置より求めた格子定数c、a及びc/aを表3に示す。又得られた焼結磁石をパーミアンス係数P_c=2.0;縦8.3mm×横7.0mm×長さ5.9mm(長さ方向が磁化方向)の直方体状に加工し、熱減磁率測定用試料とした。この試料の磁化方向に4.1MA/m(52kOe)の磁場を印加して着磁後、室温(25℃)で着磁方向のフラックス量(Φ 1)を測定した。次いで試料を恒温槽に入れ、120℃で1時間加熱後、室温(25℃)まで冷却し、フラックス量(Φ 2)を測定した。 Φ 1及び Φ 2より、式(2)により算出した熱減磁率を図2に示す。図2より熱減磁率が非常に小さく、耐熱性に富むことがわかる。

$$(\Phi 1 - \Phi 2) \div \Phi 1 \times 100 (\%) \quad (2)$$

【0024】

【表1】

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	Nd (%)	Pr (%)	B (%)	C (%)	Co (%)	Ga (%)	Cu (%)	Fe (%)	O (%)
実施例1	23.70	6.50	0.80	0.18	1.98	0.08	0.10	残部	0.12
比較例1	23.72	6.48	0.93	0.07	2.00	0.08	0.09	残部	0.13
比較例2	23.70	6.49	0.80	0.07	1.99	0.08	0.10	残部	0.12
比較例3	23.71	6.50	0.45	0.50	2.00	0.08	0.10	残部	0.15

【0025】

【表2】

	(BH) _{max} (kJ/m ³) (MG0e)	Br (T) (kG)	iHc (MA/m) (kOe)	密度 (Mg/m ³)
実施例1	384 48.2	1.42 14.2	1.16 14.4	7.59
比較例1	380 47.7	1.42 14.2	1.10 13.9	7.52
比較例2	325 40.8	1.39 13.9	0.67 8.5	7.58
比較例3	330 41.5	1.38 13.8	0.64 8.1	7.61

【0026】

【表3】

	c (nm)	a (nm)	c/a
実施例1	1.215	0.879	1.382
比較例1	1.219	0.880	1.386
比較例2	1.220	0.879	1.388
比較例3	1.209	0.882	1.371

【0027】（比較例1）重量％で、Nd：23.90%，Pr：6.60%，B：0.90%，C：0.03%，Co：2.00%，Ga：0.10%，Cu：0.10%及び残部：Feの主要成分組成を有する合金粗粉とした以外は実施例1と同様にして焼結磁石を作製した。組成分析結果を表1に、磁気特性及び密度の測定結果を表2に、格子定数比（c/a）の測定結果を表3に、X線回折パターンを図1に、熱減磁率を図2にそれぞれ示す。表2より比較例1の焼結磁石の(BH)_{max}及びiHcは実施例1のものよりやや低く、又図2より熱減磁率が実施例1より劣ることがわかる。

【0028】（比較例2）重量％で、Nd：23.90%，Pr：6.60%，B：0.80%，C：0.03%，Co：2.00%，Ga：0.10%，Cu：0.10%及び残部：Feの主要成分組成の合金粗粉とした以外は実施例1と同様にして焼結磁石を作製した。組成分析結果を表1に、磁気特性及び密度の測定結果を表2に、格子定数比（c/a）の測定結果を表3にそれぞれ示す。

（比較例3）重量％で、Nd：23.90%，Pr：6.60%，B：0.45%，C：0.50%，Co：2.00%，Ga：0.10%，Cu：0.10%及び残部：Feの主要成分組成の合金粗粉とした以外は実施例1と同様にして焼結磁石を作製した。組成分析結果を表1に、磁気特性及び密度の測定結果を表2に、格子定数比（c/a）の測定結果を表3

にそれぞれ示す。比較例2、3の焼結磁石はいずれも保磁力が極めて低く、実用に耐えないものである。この理由として、比較例2、3のものはBが化学量論組成（約0.90重量％）よりも不足しているため主相及びRリッチ相以外の第3相（R₂Fe₁₇相）が生成し、磁気特性が低下したことがわかった。これに対し、実施例1のものは化学量論組成よりもBが不足している分を溶解時点で添加したCが補い、R₂(Fe, Co)₁₄(B, C)相を形成し、もって磁気特性の改善がなされたことがわかった。関連した検討から実施例1の磁石の高いiHcは異方性磁界H_aの増加によることがわかった。

【0029】（実施例2）実施例1で作製した板厚約0.3mmのストリップキャスト合金を真空炉中に装入し、約6.7Pa（5×10⁻²Torr）の真空中で1000℃×2時間熱処理後室温まで冷却した。この熱処理済みの合金を用い、以降は実施例1と同様にして粗粉化、微粉碎、スラリー化、横磁場圧縮成形、脱油、焼結及び熱処理を行い、焼結磁石を作製した。室温（20℃）の磁気特性及び密度は下記の通りであり、実施例1に比べてiHc及び(BH)_{max}が向上していた。

(BH)_{max}：389kJ/m³（48.8MG0e）

Br：1.42T（14.2kG）

iHc：1.19MA/m（14.9kOe）

密度：7.59Mg/m³

【0030】（実施例3、4）ストリップキャスト合金の溶解主要成分組成を変えた以外は実施例1と同様にして、最終的に表4の組成の焼結磁石を作製し、室温（20℃）の磁気特性及び密度を測定した。結果を表5に示す。

【0031】

【表4】

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	Nd (%)	Pr (%)	Dy (%)	B (%)	C (%)	Co (%)	Ga (%)	Cu (%)	Fe (%)	O (%)
実施例 4	24.00	6.50	—	0.78	0.20	—	0.08	—	残部	0.13
実施例 5	23.10	6.40	1.00	0.85	0.22	2.00	0.08	0.10	残部	0.14

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【0032】

【表5】

	(BH) _{max} (kJ/m ³) (MG0e)	Br (T) (kG)	iHc (MA/m) (kOe)	密度 (Mg/m ³)
実施例 4	388 48.8	1.42 14.2	1.18 14.8	7.59
実施例 5	357 44.9	1.37 13.7	1.31 16.5	7.62

【0033】表4、5より実施例1対比でCo及びCuを含有しない実施例4の焼結磁石で実施例1と同等の室温の磁気特性が得られた。又Dyを含有する実施例5の焼結磁石で350.1kJ/m³ (44MG0e) 超の(BH)_{max}及び1.1MA/m (14kOe) 超のiHcが得られることがわかる。

【0034】

【発明の効果】以上記述の通り、本発明によれば、主要成分として所定量のCを含有した高C含有型であっても高い磁気特性を有するR-T-B-C系統焼結磁石（但しRは希土類元素の少なくとも1種であり、TはFe、又はFe及びCoである）及びその製造方法を提供することができる。又高C含有量のR-T-B系統焼結磁石のス

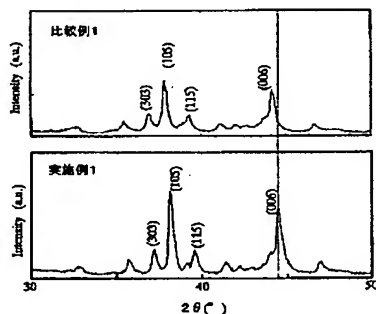
クラップを用いてリメルトしR-T-B-C系統焼結磁石用原料合金をリサイクルするに際し、許容C含有量の上限が従来のR-T-B系統焼結磁石よりも引き上げられる分リメルトにおけるスクラップ比率を増大できるというコストメリットも得られる。

【図面の簡単な説明】

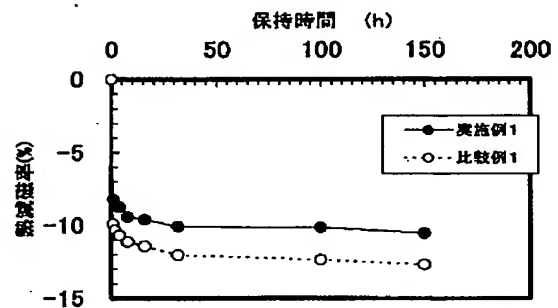
【図1】本発明の焼結磁石のX線回折パターンの一例を示す図であり、縦軸はX線回折強度（任意スケール）、横軸は回折角度である。

【図2】本発明の焼結磁石の熱減磁率の一例を示す図である。

【図1】



【図2】



フロントページの続き

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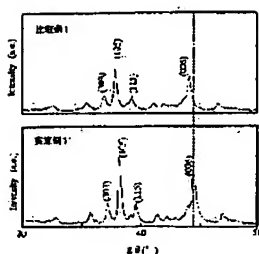
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(21)Application number : 2001- (71)Applicant : HITACHI METALS LTD
087146

(22)Date of filing : 26.03.2001 (72)Inventor : TOKORO HISATO

(54) R-T-B-C BASED SINTERED MAGNET AND PRODUCTION METHOD
THEREFOR



(57)Abstract:

PROBLEM TO BE SOLVED: To provide a high performance R-T-B-C based sintered magnet (wherein R is at least one rare earth element; and T is Fe, or Fe and Co), and a production method therefor.

SOLUTION: An R-T-B-C based alloy having the main componential composition of, by weight, 28 to 33% R (wherein R is at least one rare earth element) and 0.9 to 1.1% B+C (wherein the content of B is 0.6 to 0.9%, and the content of C is 0.15 to 0.3) and the balance T (wherein T is Fe, or Fe and Co) and having a main phase consisting of an R₂T₁₄ (B, C) phase is pulverized. The obtained fine powder is recovered into a nonoxidizing solution consisting of oil such as mineral oil and at least one kind of lubricant selected from the monovalent alcohol ester of a polybasic acid, the fatty acid ester of polyhydric alcohol, and their derivatives, and is then subjected to forming, degreasing, sintering, and heat treatment.

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CLAIMS

[Claim(s)]

[Claim 1] By weight %, R (however, R is at least one sort of rare earth elements):28 - 33%, B+C: 0.9-1.1% (however, it is B:0.6 - 0.9%, and is C:0.15 - 0.3%), And the remainder : It has the major component presentation of T (however, T is Fe, or Fe and Co). At least one sort of oils which pulverize the R-T-B-C system alloy which makes the main phase R₂T₁₄ (B, C) phase in mean particle diameter of 1-10 micrometers in a non-oxidizing atmosphere, and are chosen from mineral oil, synthetic oil, and vegetable oil in the obtained fines, It collects and slurs in the non-oxidizing quality liquid which consists of at least one sort of lubricant chosen from the monohydric alcohol ester of polybasic acid, the

fatty acid ester of polyhydric alcohol, and those derivatives. Subsequently, the manufacture approach of the R-T-B-C system sintered magnet characterized by fabricating said slurry, and deoiling, sintering and heat-treating the acquired Plastic solid.

[Claim 2] the mixing ratio of at least one sort of oils chosen from mineral oil, synthetic oil, and vegetable oil, and at least one sort of lubricant chosen from the monohydric alcohol ester of polybasic acid, the fatty acid ester of polyhydric alcohol, and those derivatives -- the manufacture approach of a R-T-B-C system sintered magnet according to claim 1 that a rate is the 99.7-99.99 weight section:0.3 - 0.01 weight section.

[Claim 3] By weight %, R (however, R is at least one sort of rare earth elements):28 - 33%, B+C: 0.9-1.1% (however, it is B:0.6 - 0.9%, and is C:0.15 - 0.3%), And the remainder : The alloy molten metal which has the major component presentation of T (however, T is Fe, or Fe and Co) is solidified by the strip cast method. The R-T-B-C system alloy which is 0.05-3mm of board thickness whose diameter of average crystal grain of the direction of a minor axis of said main phase is 3-20 micrometers while becoming substantial is obtained from the R2T 14 (B, C) main phase and R rich phase. Next, at least one sort of oils which coarse-powder-ize this alloy, pulverize in a non-oxidizing atmosphere subsequently to the mean particle diameter of 1-10 micrometers, and are chosen from mineral oil, synthetic oil, and vegetable oil in the obtained fines, It collects and slurs in the non-oxidizing quality liquid which consists of at least one sort of lubricant chosen from the monohydric alcohol ester of polybasic acid, the fatty acid ester of polyhydric alcohol, and those derivatives. Subsequently, the manufacture approach of the R-T-B-C system sintered magnet characterized by fabricating said slurry, and deoiling, sintering and heat-treating the acquired Plastic solid.

[Claim 4] the mixing ratio of at least one sort of oils chosen from mineral oil, synthetic oil, and vegetable oil, and at least one sort of lubricant chosen from the monohydric alcohol ester of polybasic acid, the fatty acid ester of polyhydric

alcohol, and those derivatives -- the manufacture approach of a R-T-B-C system sintered magnet according to claim 3 that a rate is the 99.7-99.99 weight section:0.3 - 0.01 weight section.

[Claim 5] The manufacture approach of the R-T-B-C system sintered magnet according to claim 3 or 4 which heat-treats the R-T-B-C system alloy which solidified by the strip cast method and was obtained at 800-1100 degrees C, and subsequently coarse-powder-izes it.

[Claim 6] The R-T-B-C system sintered magnet characterized by having the major component presentation of R (however, R is at least one sort of rare earth elements):28 - 33%, B+C:0.9-1.1% (however, it is B:0.6 - 0.9%, and is C:0.15 - 0.3%), and remainder:T (however, T is Fe, or Fe and Co) by weight %, and making R₂T₁₄ (B, C) phase into the main phase.

[Claim 7] The lattice-constant ratio of the R₂T₁₄ (B, C) main phase: The R-T-B-C system sintered magnet according to claim 6 whose c/a (however, c is the lattice constant of the direction of uniaxial anisotropy of *****, and a is the remaining lattice constants of two sides) is 1.375-1.385.

[Claim 8] They are R:28 - 33%, and B+C:0.9-1.1% (however, it is B:0.6 - 0.9%) at weight %. M:0.01 - 0.3% (however, M is at least one sort chosen from the group which consists of Cu, aluminum, Ga, Nb, and Mn) and remainder:T (however, T is Fe and Co) which are C:0.15 - 0.3% Co: The R-T-B-C system sintered magnet according to claim 6 or 7 which has the major component presentation of being 0.5-5%.

[Claim 9] By weight %, R (however, R is at least one sort of rare earth elements):28 - 32%, B+C: 0.9-1.1% (however, it is B:0.6 - 0.9%, and is C:0.15 - 0.3%), And the remainder : It has the major component presentation of T (however, T is Fe, or Fe and Co). The lattice-constant ratio of the R₂T₁₄ (B, C) main phase: c/a (however, c is the lattice constant of the direction of uniaxial anisotropy of *****) It is the R-T-B-C system sintered magnet which are 1.375-1.385. a -- the remaining lattice constants of two sides -- it is -- The R-T-B-C system sintered magnet characterized by for the amount of content oxygen per

unit weight of said R-T-B-C system sintered magnet being 0.3 or less % of the weight, and sintered compact consistencies being three or more 7.56 Mg/m.

[Claim 10] It is R (however, R is at least two sorts of rare earth elements, and contains Nd and Dy indispensable) at weight %. Dy content is 0.3 - 15% -- 28-32% and B+C:0.9-1.1% (however, it is B:0.6 - 0.9%) And it is C:0.15 - 0.3%, it has the major component presentation of remainder: T (however, T is Fe, or Fe and Co). The lattice-constant ratio of the R₂T₁₄ (B, C) main phase: c/a (however, c is the lattice constant of the direction of uniaxial anisotropy of *****)

It is the R-T-B-C system sintered magnet which are 1.375-1.385. a -- the remaining lattice constants of two sides -- it is -- The R-T-B-C system sintered magnet characterized by for the amount of content oxygen per unit weight of said rare earth sintered magnet being 0.3 or less % of the weight, and sintered compact consistencies being three or more 7.60 Mg/m.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the sintered magnet which

consists of a R-T-B-C system alloy (however, R is at least one sort of rare earth elements, and T is Fe, or Fe and Co), and its manufacture approach.

[0002]

[Description of the Prior Art] A Nd-Fe-B system sintered magnet carries out coarse grinding of the Nd-Fe-B system alloy of a predetermined presentation, is manufactured by pulverizing, fabricating impalpable powder with a mean particle diameter of 1-10 micrometers obtained among a magnetic field, sintering it, and subsequently, heat-treating it in the inert gas of N₂ grade, and is used abundantly in the various magnet application product fields.

[0003] In order to raise the magnetic properties of a Nd-Fe-B system sintered magnet, it is supposed that the thing low as much as possible of the content of C which is generally an unescapable impurity element is good. The manufacture approach of the high performance Nd-Fe-B system sintered magnet which mixes and slurs the raw material impalpable powder for Nd-Fe-B system sintered magnets, mineral oil, or synthetic oil in the patent No. 2731337 official report, subsequently carries out a wet compaction to it, and deoils, sinters and heat-treats the acquired Plastic solid is indicated. While an oxygen content is notably reduced compared with the former according to this approach, a degree of sintering can improve, and the high residual magnetic flux density Br, high maximum energy product (BH) max, and the Nd-Fe-B system sintered magnet that has the high proper coercive force iHc can be manufactured. These high magnetic properties perform deoiling (deC) processing before sintering so that the above-mentioned oil may not remain as much as possible in a Plastic solid, and they are attained by reducing C content of the Nd-Fe-B system sintered magnet finally obtained to 0.1 or less % of the weight.

[0004] JP,9-17677,A 12 to R(however, R is at least one sort of rare earth elements containing Y)18 atom %, Six to B+C=10 atom % (however, they are B:2 to 6 atom %, and C:4 to 8 atom %), The alloy molten metal which consists of the remainder Fe (however, the one section of Fe can be permuted in one sort of Co and nickel, or two sorts), and an unescapable impurity by the strip casting

method with sheet metal of 0.03-10mm of board thickness After casting to the cast piece which has the organization which R rich phase divided into 10 micrometers or less minutely, Into coarse-grinding powder with an average grain size of 10-500 micrometers which carried out coarse grinding of this cast piece, and obtained it, carry out addition mixing 0.02 to 5.0% of the weight, and a fluid lubrication agent or solid lubricant is pulverized. It is filled up with impalpable powder with a mean particle diameter of 1-10 micrometers obtained in mold at pack density 1.4 - 3.5 Mg/m³. Shaping behind orientation, sintering, and the method of manufacturing the R-Fe-B-C system permanent magnet ingredient which was excellent in corrosion resistance by carrying out aging treatment are indicated applying the pulse magnetic field more than 795.8 kA/m (10kOe) momentarily. However, a presentation differs from the sintered magnet of this invention in respect of the high C content [ingredient / this / permanent magnet] C:4 to 8 atom %. If an example is given, the permanent magnet of the presentation 1 of Table 1 of JP,9-17677,A has the presentation of Nd:12.8 atom %, Dy:1.5 atom %, Co:10 atom %, B:3.2 atom %, C:4.4 atom %, and Fe:68.1 atom % (Nd:28.2 % of the weight, Dy:3.7 % of the weight, Co:9.0 % of the weight, B:0.5 % of the weight, C:0.8 % of the weight, and Fe:57.9 % of the weight). Moreover, it oxidizes, is easy, it has and the R2T from examination of this invention persons 14 (B, C) main phase is oxidation. It turned out that the rate of the R2T 14 (B, C) main phase ratio falls, and it is stabilized in a room temperature, and it is (BH) max more than 350.1 kJ/m³ (44MGOe), and iHc more than 1.1 MA/m (14kOe) cannot be obtained.

[0005] In the R-Fe-B-C system alloy magnet (however, R is at least one sort of the rare earth elements containing Y), as for the patent No. 2739502 official report, each of the magnetic crystal grain of this alloy is covered by the oxidation-resistant protective coat, and this oxidation-resistant protective coat is indicating the outstanding thing of the oxidation resistance that 0.1 - 16 atom % of whose is C on the parenchyma of the alloy element which constitutes this magnetic crystal grain, including all. However, as compared with the sintered magnet of this

invention, the R-Fe-B-C system alloy magnet of each example of this official report is a high R-low B-quantity C presentation, or is the thing of a low R-low B-quantity C presentation. If an example is given, the magnet presentation of the example 1 of the patent No. 2739502 official report Nd:18 atom %, Fe:71 atom %, B:1 atom %, and C:10 atom % (Nd: – 38.8% of the weight) They are Fe:59.3 % of the weight, B:0.1 % of the weight, and C:1.8 % of the weight. The magnet presentations of an example 11 are Nd:10 atom %, Fe:79 atom %, B:1 atom %, and C:10 atom % (Nd:24.1 % of the weight, Fe:73.7 % of the weight, B:0.2 % of the weight, and C:2.0 % of the weight). The magnetic properties furthermore acquired were very low, and when these presentations were chosen from examination of this invention persons, in a room temperature, it is (BH) max more than 350.1 kJ/m³ (44MGOe), and it turned out that iHc more than 1.1MA (14kOe)/m cannot be obtained.

[0006]

[Problem(s) to be Solved by the Invention] Therefore, even if the technical problem which this invention tends to solve is the high C content mold which contained C of the specified quantity as a major component, it is offering the R-T-B-C system sintered magnet (however, R's being at least one sort of rare earth elements, and T's being Fe, or Fe and Co) which has high magnetic properties, and its manufacture approach.

[0007]

[Means for Solving the Problem] The manufacture approach of the R-T-B-C system sintered magnet of this invention which solved the above-mentioned technical problem By weight %, R (however, R is at least one sort of rare earth elements):28 - 33%, B+C: 0.9-1.1% (however, it is B:0.6 - 0.9%, and is C:0.15 - 0.3%), And the remainder : It has the major component presentation of T (however, T is Fe, or Fe and Co). At least one sort of oils which pulverize the R-T-B-C system alloy which makes the main phase R₂T₁₄ (B, C) phase in mean particle diameter of 1-10 micrometers in a non-oxidizing atmosphere, and are chosen from mineral oil, synthetic oil, and vegetable oil in the obtained fines, It

collects and slurs in the non-oxidizing quality liquid which consists of at least one sort of lubricant chosen from the monohydric alcohol ester of polybasic acid, the fatty acid ester of polyhydric alcohol, and those derivatives. Subsequently, it is characterized by fabricating said slurry, and deoiling, sintering and heat-treating the acquired Plastic solid. On the occasion of manufacture of the R-T-B-C system sintered magnet of this invention, it is necessary to make the addition stage of C into the phase the dissolution phase of an alloy, or before the dissolution (for C of the specified quantity to be made to contain beforehand in the raw material for the dissolution). After this reason's ingoting the R-T-B system alloy which makes the main phase the R2T14 B phase containing C of unescapable impurity level, for example, in the sintered magnet which adds C of the specified quantity in forms, such as carbon black, at the time of coarse grinding or pulverizing, subsequently fabricates among a magnetic field, and sinters, and it comes to heat-treat It is because the inclination for C added at the time of coarse grinding or pulverizing to hardly be taken in into the R2T14B main phase, but for C to be condensed in R rich phase is remarkable and the improvement effectiveness of the magnetic properties by R2T14 (B, C) phase cannot be expected.

[0008] Moreover, the manufacture approach of the R-T-B-C system sintered magnet of this invention By weight %, R (however, R is at least one sort of rare earth elements):28 - 33%, B+C: 0.9-1.1% (however, it is B:0.6 - 0.9%, and is C:0.15 - 0.3%), And the remainder : The alloy molten metal which has the major component presentation of T (however, T is Fe, or Fe and Co) is solidified by the strip cast method. The R-T-B-C system alloy which is 0.05-3mm of board thickness whose diameter of average crystal grain of the direction of a minor axis of said main phase is 3-20 micrometers while becoming substantial is obtained from the R2T 14 (B, C) main phase and R rich phase. Next, at least one sort of oils which coarse-powder-ize this alloy, pulverize in a non-oxidizing atmosphere subsequently to the mean particle diameter of 1-10 micrometers, and are chosen from mineral oil, synthetic oil, and vegetable oil in the obtained fines, It collects

and slurs in the non-oxidizing quality liquid which consists of at least one sort of lubricant chosen from the monohydric alcohol ester of polybasic acid, the fatty acid ester of polyhydric alcohol, and those derivatives. Subsequently, it is characterized by fabricating said slurry, and deoiling, sintering and heat-treating the acquired Plastic solid.

[0009] the mixing ratio of at least one sort of oils chosen from mineral oil, synthetic oil, and vegetable oil in the manufacture approach of the R-T-B-C system sintered magnet of this invention, and at least one sort of lubricant chosen from the monohydric alcohol ester of polybasic acid, the fatty acid ester of polyhydric alcohol, and those derivatives -- since the amount of preferred orientation of a Plastic solid will improve if a rate is made into the 99.7-99.99 weight section:0.3 - 0.01 weight section, it is desirable, and it has and Br and (BH) max improve. If a mixed ratio separates from said specific range, the improvement effectiveness in the amount of preferred orientation of a Plastic solid cannot be acquired, or R forms rare earth carbide by the amount of C supplied to a grain boundary phase increasing, and poor sintering is invited.

[0010] Br of the R-T-B-C system sintered magnet which becomes the sharp fines of the diameter distribution of a grain which heat-treat the R-T-B-C system alloy obtained by carrying out the strip cast in the manufacture approach of the R-T-B-C system sintered magnet of this invention at 800-1100 degrees C in a substantial vacuum or an inert gas ambient atmosphere, subsequently coarse-powder-ize it, and pulverize it, has, and is finally obtained, (BH) max, and the square shape nature of a demagnetization curve can be improved.

[0011] The R-T-B-C system sintered magnet of this invention has the major component presentation of R (however, R is at least one sort of rare earth elements):28 - 33%, B+C:0.9-1.1% (however, it is B:0.6 - 0.9%, and is C:0.15 - 0.3%), and remainder:T (however, T is Fe, or Fe and Co) by weight %, and is characterized by making R₂T₁₄ (B, C) phase into the main phase. the R-T-B-C system sintered magnet of this invention -- setting -- the lattice constant ratio of the R₂T₁₄ (B, C) main phase -- high Br and high (BH) max can be obtained at

the time of : $c/a=1.375-1.385$ (however, c is the lattice constant of the direction of uniaxial anisotropy of *****, and a is the remaining lattice constants of two sides). Moreover, it sets to the R-T-B-C system sintered magnet of this invention, and they are R:28 - 33%, and B+C:0.9-1.1% (however, it is B:0.6 - 0.9%) at weight %. M:0.01 - 0.3% (however, M is at least one sort chosen from the group which consists of Cu, aluminum, Ga, Nb, and Mn) and remainder:T (however, T is Fe and Co) which are C:0.15 - 0.3% Co: Since magnetic properties and corrosion resistance can be improved if the major component presentation of being 0.5-5% is chosen, it is desirable.

[0012] The R-T-B-C system sintered magnet of this invention by weight % Moreover, R (however, R is at least one sort of rare earth elements):28 - 32%, B+C: 0.9-1.1% (however, it is B:0.6 - 0.9%, and is C:0.15 - 0.3%), And the remainder : It has the major component presentation of T (however, T is Fe, or Fe and Co). The lattice-constant ratio of the R2T 14 (B, C) main phase: c/a (however, c is the lattice constant of the direction of uniaxial anisotropy of *****) a -- the remaining lattice constants of two sides -- it is -- it is characterized by being the R-T-B-C system sintered magnet which are 1.375-1.385, and for the amount of content oxygen per unit weight of said R-T-B-C system sintered magnet being 0.3 or less % of the weight, and sintered compact consistencies being three or more 7.56 Mg/m.

[0013] Moreover, the R-T-B-C system sintered magnet of this invention is R (however, R is at least two sorts of rare earth elements, and contains Nd and Dy indispensable) at weight %. Dy content is 0.3 - 15% -- :28-32% and B+C:0.9-1.1% (however, it is B:0.6 - 0.9%) And it is C:0.15 - 0.3%, it has the major component presentation of remainder:T (however, T is Fe, or Fe and Co). The lattice-constant ratio of the R2T 14 (B, C) main phase: c/a (however, c is the lattice constant of the direction of uniaxial anisotropy of *****) a -- the remaining lattice constants of two sides -- it is -- it is characterized by for the amount of oxygen which is the R-T-B-C system sintered magnet which are 1.375-1.385, and is contained in per unit weight of said rare earth sintered magnet being 0.3 or

less % of the weight, and sintered compact consistencies being three or more 7.60 Mg/m.

[0014] the grain boundary phase which controlled oxide-izing and carbide-izing of R component, and was rich in R component in the R-T-B-C system sintered magnet of this invention -- securing -- the rate of the R₂T₁₄ (B, C) main phase ratio -- as much as possible -- height -- having -- Br and (BH) -- in order to raise max and iH_c, it is more desirable to **** the amount of content oxygen to 0.2 or less % of the weight, and it is still more desirable to carry out to 0.18 or less % of the weight.

[0015]

[Embodiment of the Invention] The reason for presentation limitation of the R-T-B-C system sintered magnet of this invention is explained below. Only describing it as % below shall mean weight %.

[0016] 28 - 33% of the amount of R is desirable. In order to provide good corrosion resistance and to raise (BH) max, 28 - 32% of the amount of R is more desirable, and is desirable. [especially 28 - 31% of] iH_c to which the amount of R is equal to practical use at less than 28% cannot be obtained, but the fall of Br and (BH) max becomes remarkable by ** 33%. Except for an unescapable R component, the practicality in R=Nd+Dy, Nd+Dy+Pr, Nd+Pr, or Pr+Dy is high. In order to give the coercive force (thermal resistance) which is equal to practical use, 0.3 - 10% of Dy content is desirable, and it is more desirable to make it to 0.5 - 8%. Dy content cannot acquire the improvement effectiveness of coercive force at less than 0.3%, but Br and (BH) max fall greatly by ** 10%.

[0017] B and C are essential elements, and 0.9 - 1.1% of an amount (B+C) is desirable, and is more desirable. [0.95 - 1.1% of] 0.6 - 0.9% of the amount of B is desirable, and is more desirable. [0.65 - 0.85% of] 0.15 - 0.3% of the amount of C is desirable, and is more desirable. [0.18 - 0.28% of] At less than 0.6%, Br and iH_c fall [the amount of B / the amount of C] greatly with generation of R₂Fe₁₇ intermetallic compound also in specific above-mentioned content within the limits. Moreover, room for C to almost enter [the amount of B] into the main

phase by ** 0.9% is lost, and since it is condensed by the grain boundary phase as an impurity, Br and iHc fall greatly. Since too little [at less than 0.15% / the amount of generation of R2T14 (B, C) phase], the amount of C cannot acquire the improvement effectiveness in magnetic properties by R2T14 (B, C) phase as a matter of fact, but by **, C is condensed by the grain boundary phase 0.3%, and while inviting poor sintering, magnetic properties fall rapidly. That is, the high magnetic properties which R2T14 (B, C) phase generated as a main phase, had at the time of the above-mentioned amount range of specification (B+C), and were not acquired in the conventional high C content mold Nd-Fe-B sintered magnet can be acquired.

[0018] Magnetic properties and corrosion resistance can be raised by containing at least one sort of elements M chosen from the group which consists of Cu, aluminum, Ga, Nb, and Mn 0.01 to 0.3%. By containing aluminum 0.01 to 0.3%, iHc improves and corrosion resistance is improved. However, by **, the fall of Br becomes remarkable 0.3%, and aluminum content cannot acquire the effectiveness which raises iHc and corrosion resistance at less than 0.01%. More desirable aluminum content is 0.05 - 0.3%. Although iHc improves notably by containing Ga 0.01 to 0.3%, by **, the fall of Br becomes large 0.3%, and Ga content cannot acquire the effectiveness which raises iHc at less than 0.01%. More desirable Ga content is 0.05 - 0.3%. Although corrosion resistance and iHc improve by containing Cu 0.01 to 0.3%, by **, the fall of Br becomes remarkable 0.3%, and Cu content cannot acquire the effectiveness which raises corrosion resistance and iHc at less than 0.01%. More desirable Cu content is 0.05 - 0.3%.

[0019] By containing Co 0.5 to 5%, corrosion resistance improves, the Curie point goes up and has and thermal resistance improves. However, a Fe-Co phase with Co content harmful to magnetic properties in 5% ** generates, and Br and iHc fall greatly. Moreover, Co content cannot acquire the effectiveness of improving corrosion resistance and thermal resistance, at less than 0.5%. Moreover, when 0.5 - 5% and Cu are contained for Co 0.01 to 0.3%, the effectiveness that the tolerance of the second heat treatment temperature which

can obtain iH_c of the room temperature more than 1.1 MA/m (14kOe) spreads can be acquired, and it is especially desirable.

[0020] 0.3% or less of the amount of oxygen contained is desirable, is more desirable, and is desirable. [especially 0.18% or less of] [0.2% or less of]

As a result of the rate of the R2T 14 (B, C) main phase ratio increasing and being able to raise a sintered compact consistency to abbreviation theoretical density by reducing an oxygen content to 0.3% or less, in a room temperature, it is (BH) max more than 350.1 kJ/m³ (44MGOe), and it is stabilized and iH_c more than 1.1 MA/m (14kOe) can be obtained. A sintered compact consistency becomes three or more 7.56 Mg/m for example, with a Nd-Pr-T-B-C system sintered magnet, and becomes three or more 7.60 Mg/m with a Nd-Dy-T-B-C system sintered magnet. Moreover, since good corrosion resistance and high magnetic properties are provided, it is desirable to make nitrogen volume contained is 0.15% or less, and it is more desirable to make it to 0.002 - 0.15%. The fall of Br will become remarkable if nitrogen volume exceeds 0.15%.

[0021] The R-T-B-C system sintered magnet of this invention is the following, and can be made and manufactured. First, the strip cast of the R-T-B-C system alloy molten metal adjusted to the predetermined presentation is carried out by the single rolling method or the congruence rolling method, it is board thickness: 0.05-3mm sheet metal, and the R-T-B-C system alloy which becomes substantial is obtained from the R2T 14 (B, C) main phase and detailed R rich phase. It is about 3-20 micrometers, and the diameter of average crystal grain of the direction of a minor axis of the R2T 14 (B, C) main phase in this alloy is an organization without αFe , and it can have it and it can obtain the R-T-B-C system sintered magnet of high magnetic properties. It is desirable to perform heat treatment which heats said sheet metal alloy in an inert gas ambient atmosphere for 800-1100 degree-Cx 0.5 to 10 hours if needed. By this heat treatment, the particle size distribution of pulverizing powder becomes Sharp, and can raise Br and (BH) max. In addition, the effectiveness of heat treatment of heat treatment conditions less than [800 degree-Cx0.5 hour] is not accepted,

but problems, such as a presentation gap by oxidation, are produced in ** for 1100 degree-Cx 10 hours. C in said sheet metal-like R-T-B-C system alloy is incorporated in the main phase. This is because C is incorporated in R2T14 B phase through the 800 degrees C - 1100 degrees C temperature field which R2T14C forms in stability a process until it is cooled by the room temperature from the melting temperature of a R-T-B-C system alloy. That is, if B makes C of optimum dose exist by stoichiometric composition contrast of R2T14B in the R-T-B system alloy molten metal adjusted to the presentation which run short a little, R2T14 (B, C) phase will be generated, and it will be thought that high magnetic properties are discovered. Next, occlusion of the hydrogen is carried out to said sheet metal-like R-T-B-C system alloy, spontaneous disintegration is carried out, and this is coarse-powder-ized after performing dehydrogenation treatment subsequently. Subsequently, it pulverizes. With the jet mill which uses inert gas as tumbling media, an oxygen density pulverizes pulverizing in mean particle diameter of 1-10 micrometers in the inert gas ambient atmosphere below 0.01 volume % more preferably under 0.1 volume %. In this way, the obtained fines are collected and slurred in the non-oxidizing quality liquid which consists of at least one sort of oils chosen from mineral oil, synthetic oil, and vegetable oil, and at least one sort of lubricant chosen from the monohydric alcohol ester of polybasic acid, the fatty acid ester of polyhydric alcohol, and those derivatives. Subsequently, said slurry is fabricated among a magnetic field, and the acquired Plastic solid is deoiled, sintered and heat-treated. The mean particle diameter of said fines has desirable 1-10 micrometers, and its 3-6 micrometers are more desirable. By less than 1 micrometer, the efficiency of comminution falls [mean particle diameter] greatly, and the fall of iHc and Br becomes remarkable in 10-micrometer **. In order to prevent degradation of the magnetic properties by oxidation of a Plastic solid, it is desirable to save a Plastic solid in said oil from immediately after shaping to deoiling. If the temperature up of the Plastic solid is rapidly carried out from a room temperature to sintering temperature, the internal temperature of a Plastic solid rises rapidly, the oil which remains to a Plastic solid,

and the rare earth elements which constitute a Plastic solid will react, rare earth carbide will be generated, and magnetic properties will deteriorate. It is desirable to perform deoiling processing heated more than for 30 minutes with the temperature of 100-500 degrees C and below the degree of vacuum of 13.3Pa (10-1Torr) as this cure. The oil in a Plastic solid is fully removed by deoiling processing. In addition, as long as it is 100-500 degrees C, whenever [stoving temperature / of deoiling processing] does not need to be one point, and two or more points are sufficient as it. Moreover, it can deoil efficiently also by performing deoiling processing which makes the following hereafter the programming rate from a room temperature to 500 degrees C by 5-degree-C/more preferably by 10-degree-C/below by 13.3Pa (10-1Torr). A thing 350 degrees C or less has a point to the good point of deoiling and a moldability distilling fractionally as mineral oil, synthetic oil, or vegetable oil. Moreover, the thing of 10 or less cSts has the good kinematic viscosity of a room temperature, and the thing of 5 or less cSts is still more desirable.

[0022] If an X diffraction is carried out in $2\theta=30-50$ degree by law 2 θ/θ by making CuK alpha rays into a line source to the R-T-B-C system sintered magnet of this invention, the strong X diffraction peak from a field (105) and a field (006) can be acquired. Spacing d ($d= X\text{-ray wavelength}/(2\sin\theta)$) and indices of crystal plane for which it asked from these two x line diffraction peak location (2θ) can be substituted for a formula (1), and it can ask for the lattice constants a and c of R2T14 B phase (tetragonal phase).

$$1/d^2=(h^2+k^2)/a^2+l^2/c^2 \quad (1)$$

[0023]

[Example] Hereafter, this invention is not limited by these examples although an example explains this invention to a detail.

By weight %, it has the major component presentation of Nd:23.90%, Pr:6.60%, B:0.80%, C:0.18%, Co:2.00%, Ga:0.10%, Cu:0.10%, and remainder:Fe.

(Example 1) The diameter of average crystal grain of about 0.3mm of board thickness and the direction of a minor axis coarse-powder-ized the strip cast alloy

which is 3 micrometers, and carried out jet mill pulverizing in the nitrogen-gas-atmosphere and subsequently to about 1 ppm (volume ratio) of oxygen densities adjusted. Fines with a mean particle diameter of 4.0 micrometers obtained were collected and slurred in mineral oil (the Idemitsu Kosan make, trade name: Idemitsu super sol PA-30), without touching atmospheric air in this nitrogen-gas-atmosphere and. Mean particle diameter was measured with the laser diffraction mold particle-size-distribution measuring device (trade name: HEROSU Rhodes) made from Sympatec. Next, the methyl oleate of the specified quantity was added to the slurry, and it mixed with the agitator. The combination items of a slurry were taken as said fines:70 weight section, the mineral oil:29.9 weight section, and the methyl oleate:0.10 weight section. Next, a slurry is injected into the cavity of a compression mold and, subsequently they are orientation magnetic-field-strength:1.0 MA/m (13kOe) and compacting pressure. : Horizontal magnetic field compression molding was carried out on condition that 98MPa (1.0 ton/cm²), and the 15mmx25mmx10mm rectangular parallelepiped-like Plastic solid was acquired. Next, on the degree of vacuum of about 66.5Pa (5x10⁻¹Torr), and 200-degree C conditions, the Plastic solid was heated for 3 hours, and was deoiled, and, subsequently the temperature up was carried out to 1050 degrees C in this ambient atmosphere, and subsequently, at 1050 degrees C, it sintered for 2 hours and cooled to the room temperature after that. Next, the first heat treatment which heats a sintered compact at 900 degrees C for 2 hours, and subsequently quenches it to a room temperature in an argon ambient atmosphere was performed. Next, it heated at 500 degrees C in the argon ambient atmosphere for 1 hour, and the second heat treatment subsequently cooled to a room temperature was performed, and the sintered magnet of this invention was obtained. The component-analysis result of the obtained sintered magnet is shown in Table 1. Moreover, after processing the obtained sintered magnet into 10mm angle and measuring a consistency, the result of having measured the magnetic properties of a room temperature (20 degrees C) is shown in Table 2. Moreover, the obtained sintered magnet was processed into

7mm angle, the pulse magnetic field of 11.9 MA/m (150kOe) was impressed in the room temperature (20 degrees C), and magnetization measurement was performed. It was $B_r/(4\pi I) \text{ max} = 97.2\%$ which calculated the maximum $(4\pi I) \text{ max}$ of B_r and magnetization by this measurement, and was computed.

Moreover, the X diffraction (line source: CuK alpha rays) was performed in the following ways to the obtained sintered magnet. The sintered magnet obtained so that a field perpendicular to the anisotropy grant direction might turn into a measuring plane was set, and $2\theta=30\sim50$ degree was scanned by law $2\theta/\theta$. A result is shown in drawing 1 . Only the X diffraction pattern as $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase with the same all was accepted. Contrast with the X diffraction pattern (inside of drawing 1) of the example 1 of a comparison mentioned later shows that the X diffraction peak of the field (006) of the sintered magnet of an example 1 has shifted to a side whenever [angle-of-elevation]. This is a proof added C indicates it to be to exist in the main phase, and the diffraction pattern in drawing 1 expresses $2(\text{Nd, Pr})(\text{Fe, Co})_{14}(\text{B, C})$ phase. (006) The lattice constants c and a and c/a which were calculated from the diffraction peak location of a field and a field (105) are shown in Table 3. Moreover, the obtained sintered magnet was processed with a permeance coefficient $P_c=2.0; 7.0\text{mm} \times \text{die length of } 5.9\text{mm}$ (the die-length direction is the magnetization direction) in the shape of a rectangular parallelepiped, and it considered as the heat demagnetizing-factor test sample. [8.3mm by] The magnetic field of 4.1 MA/m (52kOe) was impressed in the magnetization direction of this sample, and the amount of flux of the magnetization direction (ϕ_1) was measured at the room temperature (25 degrees C) after magnetization. Subsequently, the sample was paid to the thermostat, it cooled to the room temperature (25 degrees C) after 1-hour heating at 120 degrees C, and the amount of flux (ϕ_2) was measured. From ϕ_1 and ϕ_2 , the heat demagnetizing factor computed by the formula (2) is shown in drawing 2 . A heat demagnetizing factor is very small and drawing 2 shows that it is rich in thermal resistance.

$(\phi_1 - \phi_2) / \phi_1 \times 100 (\%)$ (2)

[0024]

[Table 1]

	Nd (%)	Pr (%)	B (%)	C (%)	Co (%)	Ga (%)	Cu (%)	Fe (%)	O (%)
實施例 1	23.70	6.50	0.80	0.18	1.98	0.08	0.10	殘部	0.12
比較例 1	23.72	6.48	0.93	0.07	2.00	0.08	0.09	殘部	0.13
比較例 2	23.70	6.49	0.80	0.07	1.99	0.08	0.10	殘部	0.12
比較例 3	23.71	6.50	0.45	0.50	2.00	0.08	0.10	殘部	0.15

[0025]

[Table 2]

	(BH) _{max} (kJ/m ³) (MGOe)	Br (T) (kG)	iHc (MA/m) (kOe)	密度 (Mg/m ³)
實施例 1	384 48.2	1.42 14.2	1.16 14.4	7.59
比較例 1	380 47.7	1.42 14.2	1.10 13.9	7.52
比較例 2	325 40.8	1.39 13.9	0.67 8.5	7.58
比較例 3	330 41.5	1.38 13.8	0.64 8.1	7.61

[0026]

[Table 3]

	c (nm)	a (nm)	c / a
實施例 1	1.215	0.879	1.382
比較例 1	1.219	0.880	1.386
比較例 2	1.220	0.879	1.388
比較例 3	1.209	0.882	1.371

[0027] (Example 1 of a comparison) By weight %, the sintered magnet was produced like the example 1 except having considered as the alloy coarse powder which has the major component presentation of Nd:23.90%, Pr:6.60%, B:0.90%, C:0.03%, Co:2.00%, Ga:0.10%, Cu:0.10%, and remainder:Fe. a component-analysis result -- Table 1 -- magnetic properties and the measurement result of a consistency -- Table 2 -- an X diffraction pattern is shown in drawing 1 , and a heat demagnetizing factor is shown for the measurement result of a lattice constant ratio (c/a) in Table 3 at drawing 2 , respectively. Table 2 -- (BH) of the sintered magnet of the example 1 of a

comparison -- max and iH_c are a little lower than the thing of an example 1, and it turns out that a heat demagnetizing factor is inferior to drawing 2 from an example 1.

[0028] (Example 2 of a comparison) By weight %, the sintered magnet was produced like the example 1 except having considered as the alloy coarse powder of a major component presentation of Nd:23.90%, Pr:6.60%, B:0.80%, C:0.03%, Co:2.00%, Ga:0.10%, Cu:0.10%, and remainder:Fe. Magnetic properties and the measurement result of a consistency are shown in Table 2, and the measurement result of a lattice constant ratio (c/a) is shown for a component-analysis result in Table 1 in Table 3, respectively.

(Example 3 of a comparison) By weight %, the sintered magnet was produced like the example 1 except having considered as the alloy coarse powder of a major component presentation of Nd:23.90%, Pr:6.60%, B:0.45%, C:0.50%, Co:2.00%, Ga:0.10%, Cu:0.10%, and remainder:Fe. Magnetic properties and the measurement result of a consistency are shown in Table 2, and the measurement result of a lattice constant ratio (c/a) is shown for a component-analysis result in Table 1 in Table 3, respectively. Each sintered magnet of the examples 2 and 3 of a comparison has very low coercive force, and does not bear practical use. It turned out that the 3rd phase other than the main phase and R rich phase (R_2Fe_{17} phase) generated as this reason since B was insufficient for the thing of the examples 2 and 3 of a comparison from stoichiometric composition (about 0.90 % of the weight), and magnetic properties fell. On the other hand, it turned out that C which it is at the dissolution time and was added compensated with the thing of an example 1 the part which runs short of B rather than stoichiometric composition, it formed and had $R_2(Fe, Co)_{14}(B, C)$ phase, and the improvement of magnetic properties was made. A related examination showed that iH_c with the expensive magnet of an example 1 was based on the increment in an anisotropy field H_a .

[0029] (Example 2) The strip cast alloy of about 0.3mm of board thickness produced in the example 1 was inserted in all over the vacuum furnace, and it

cooled to the after [1000 degree-Cx 2 hour heat treatment] room temperature with the degree of vacuum of about 6.7Pa (5x10⁻²Torr). Using this heat treated alloy, coarse-powder-izing, pulverizing, slurring, horizontal magnetic field compression molding, deoiling, sintering, and heat treatment were performed like the example 1, and the sintered magnet was produced henceforth. The magnetic properties and the consistency of a room temperature (20 degrees C) are as follows, and iHc and its (BH) max were improving compared with the example 1. (BH)_{max}:389kJ/m³(48.8MGOe)

Br:1.42T (14.2kG

iHc: 1.19 MA/m consistency (14.9kOe): 7.59 Mg/m³ [0030] (Examples 3 and 4)

Except having changed the dissolution major component presentation of a strip cast alloy, like the example 1, finally the sintered magnet of a presentation of Table 4 was produced, and the magnetic properties and the consistency of a room temperature (20 degrees C) were measured. A result is shown in Table 5.

[0031]

[Table 4]

	Nd (%)	Pr (%)	Dy (%)	B (%)	C (%)	Co (%)	Ga (%)	Cu (%)	Fe (%)	O (%)
实施例 4	24.00	6.50	—	0.78	0.20	—	0.08	—	残部	0.18
实施例 5	23.10	6.40	1.00	0.85	0.22	2.00	0.08	0.10	残部	0.14

[0032]

[Table 5]

	(BH) _{max} (kJ/m ³) (MGOe)	Br (T) (kG)	iHc (MA/m) (kOe)	密度 (Mg/m ³)
实施例 4	388 48.8	1.42 14.2	1.18 14.8	7.59
实施例 5	357 44.9	1.37 13.7	1.31 16.5	7.62

[0033] The magnetic properties of a room temperature equivalent to an example 1 were acquired with the sintered magnet of the example 4 which does not contain Co and Cu by example 1 contrast from Tables 4 and 5. Moreover, they are 350.1 kJ/m³ (44MGOe) with the sintered magnet of the example 5 containing Dy. It turns out that (BH) max of ** and iHc of 1.1 MA/m (14kOe) ** are obtained.

[0034]

[Effect of the Invention] Above, according to this invention as description, even if it is the high C content mold which contained C of the specified quantity as a major component, the R-T-B-C system sintered magnet (however, R is at least one sort of rare earth elements, and T is Fe, or Fe and Co) which has high magnetic properties, and its manufacture approach can be offered. Moreover, it faces, and recycling the raw material alloy for R-T-B-C system sintered magnets, and the cost merit that the upper limit of a permission C content can increase the scrap ratio in part RIMERUTO which can be pulled up from the conventional R-T-B system sintered magnet is also obtained. [using the scrap of the R-T-B system sintered magnet of a quantity C content]

[Translation done.]

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is drawing showing an example of the X diffraction pattern of the sintered magnet of this invention, and an axis of ordinate is X-ray time reinforcement (arbitration scale), and an axis of abscissa is whenever [angle-of-diffraction].

[Drawing 2] It is drawing showing an example of the heat demagnetizing factor of the sintered magnet of this invention.

[Translation done.]

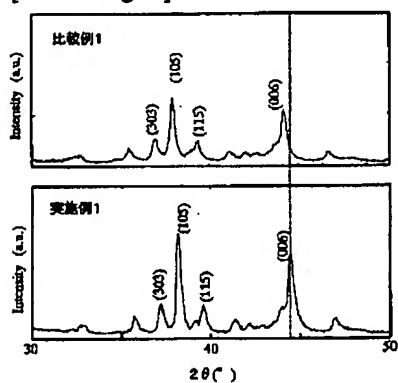
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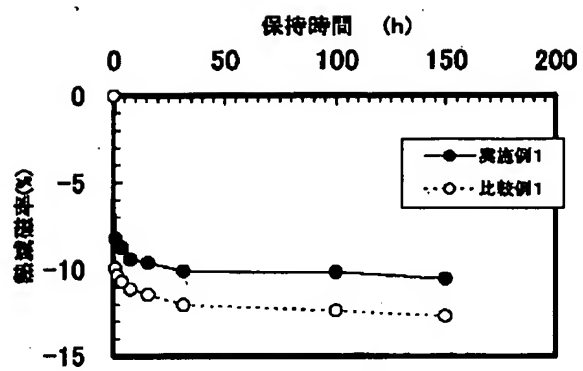
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DRAWINGS

[Drawing 1]



[Drawing 2]



[Translation done.]